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Function and Safety Tests of NOS-365 Monopropellant

by
H. Dean Mallory
Research Department
for the
Propulsion Development Department

APRIL 1977

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R. G. Freeman, III, RAdm., USN . . . . Commander

G. L. Hollingsworth . . . . Technical Director

FOREWORD

This report describes an experimental investigation, conducted during the period August 1973 through April 1976, of NOS-365 monopropellant. The work was sponsored by the Naval Air Systems Command under AirTask 350-350B/008B/4F32-363-502.

Dr. James W. Bryant has reviewed this report for technical accuracy.

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(U) *Function and Safety Tests of NOS-365 Monopropellant*, by H. Dean Mallory. China Lake, Calif., Naval Weapons Center, April 1977, 26 pp. (NWC TP 5940, publication UNCLASSIFIED.)

(U) Several non-standard function and safety tests were performed on NOS-365 monopropellant. The function tests indicated that the liquid was detonable but it may be safely used as a gun propellant if the gun chamber is shorter than a critical run-up distance. Spill tests showed vigorous fume-off when the liquid came in contact with rusty iron or desert soil warmed by the sun.

CONTENTS

INTRODUCTION AND SUMMARY

In 1965, NWC began using the impedance mirror technique to investigate detonation reaction zone effects. This experimental procedure is not yet in use by other laboratories. Data not available using standard procedures can be obtained using this technique, e.g., detonation reaction time across the wave front.[1]

The propellant development portion of the liquid propellant gun program consisted of two primary efforts: (1) the bipropellant work at NWC, China Lake, and (2) the monopropellant work at the Naval Ordnance Station (NOS), Indian Head. Most of the monopropellant tests were conducted by NOS; however, the impedance mirror tests could only be conducted at NWC. In accordance with instructions from the Naval Air Systems Command, several non-standard test sequences were devised to determine the suitability of the NOS-365 monopropellant for air applications.

Although all standard tests had indicated that NOS-365 was nondetonable, initial impedance mirror tests showed that the liquid did detonate under strong booster shock. These tests were, therefore, continued and other experimental procedures were devised based on information generated.

NOS-365 has proven to be a propellant which in our experience is somewhat unique. Its reaction time while undergoing detonation is in excess of 10 microseconds, as compared to an earlier monopropellant, nitromethane, whose reaction time is 22 nanoseconds. Although total experience is still quite limited, nanosecond reaction time appears to be typical of explosive compounds such as TNT and Composition B. The 10 microsecond reaction time of NOS-365 suggested that, even though detonable, the monopropellant could have a critical run-up distance requirement which would allow positive control of the burning phase. Briefly, a critical run-up requirement would mean that a gun chamber shorter than the run-up distance would always prevent detonation without regard to any accidental condition of ignition leading to strong initiation. Run-up test results will be detailed later in this report.

IMPEDANCE MIRROR TESTS

The impedance mirror is ordinarily a 6- by 6- by 1-inch Plexiglas block, aluminized on one side to produce an optically good mirror. The propellant to be tested is placed in contact with the aluminized side. The camera (streak or framing) observes the Plexiglas which serves as a second surface mirror. An explosive argon flash bomb, placed to give specular reflection from the mirror to the camera, provides the required high intensity illumination. A schematic diagram of the system is shown in Figure 1.

[1] H. Dean Mallory. "Detonation Reaction Time in Diluted Nitromethane," *J. Applied Physics*, Vol. 47, No. 1, (January 1976).

Operation of the mirror technique depends on several factors: (1) Plexiglas is compressible under strong shock pressure, (2) aluminized layer adheres to the plastic without rupture while the plastic surface is distorted by the impacting wave, and (3) detonation reaction zone is turbulent. Reaction zone turbulence implies that point-to-point pressure differences exist in the wave. These pressure differences produce unequal distortion at different points on the impact surface of the mirrors. In fact, they produce a pattern on the mirror which, for explosives, is as distinctly different as fingerprints are for people. This pattern in the reflective surface is a series of ridges and valleys which serve to reduce the total specular reflection from the light bomb to the camera. The reaction zone, on the photographic record, is delineated by a more or less narrow band of reduced reflectivity. After passage of the zone through the mirror, the reflectivity returns to a higher level.

Impedance mirror streak camera records show detonation reaction zone differences which depend on propellant energy. (Normal camera writing rate is 10 mm/microsecond.) The Figure 2 records show obvious differences in that the higher energy material has a shorter reaction time and a finer scale of turbulence. This effect was predicted by the Russian worker Schelkin several years before these photographs were made.[2] Even those records, such as in Figure 2a, having large reaction cells can be read with some precision and the reaction time determined. This entails using a microcomparator fitted with a photomultiplier to observe the record through a long, narrow slit. The comparator slit integrates light transmitted through the record along the distance dimension. When this transmitted light intensity is plotted against the time axis of the record, the reaction time can be read. Such a derived record is shown in Figure 3.

The above records are typical of what is expected of an explosive or detonable propellant. As has been mentioned, NOS-365 is somewhat unique; it appears to have a very long reaction time. This very uniqueness has, however, prevented complete measurements. Definitive measurements would require a small research effort to change the measuring technique; this was beyond the scope of our investigation. We have, therefore, extrapolated available information and arrived at our present position by a series of best estimates.

Two of the earliest impedance mirror records made during August 1973 are shown in Figure 4. In Figure 4a, the NOS-365 tank was 72 mm long and 127 mm in diameter. Initiation was by a 1-inch-thick, 4-inch-diameter disk of Composition B and a P-40 plane wave lens firing through a 1/8-inch sheet of aluminum. This is our standard booster which produces about 18 GPa (180 kbar) in the propellant--a very strong shock. The wave front on the right is seen to be somewhat rough, indicating the propellant was detonating with difficulty. Mirror reflectivity was greatly reduced with

[2] K. I. Schelkin. "Two Cases of Unstable Combustion," *J. Exptl. Theoret. Phys* (USSR), Vol. 36 (February 1959), p. 600; *Soviet Physics-JETP*, Vol. 36, No. 9 (August 1959), p. 416.

small increments reflective. These increments oscillated between reflection and non-reflection, indicating reaction was occurring at least until the far surface of the Plexiglas blew out.

The experiment in Figure 4b was boosted by point initiation of a tetryl pellet into a 1- by 4-inch Composition B disk without the P-40. Except at the center of the tank, the pressure was slightly lower. Here the initial shock front is curved, as it should be, and the center of the mirror remains reflective until 4.5 microseconds after blowout. The most significant observation is that reaction began at some time *after* passage of the shock front and *increased* with time at least until reflectivity was lost. From records such as this, we can say that NOS-365 detonates with difficulty and the reaction time is exceptionally long.

The next impedance mirror test series was done in March 1976. Because it was snowing during one testing day, we can be fairly sure of the propellant's upper temperature limit. The records obtained were all about as shown in Figure 5.

The Figure 5 experiment was set up using a 58 mm long, 105 mm ID propellant tank boosted by a standard P-40, Composition B booster. Notice that the wave front on the right is smoothly curved despite the tank being shorter than that of Figure 4a. The smoothly curved front is typical of a totally unreactive shock. Despite some reflectivity remaining, there are no mirror oscillations which are characteristic of points of reaction in the propellant. We could only conclude that the propellant was nondetonable. The snow during testing was a fortunate reminder that the propellant was cold. (The August 1973 tests were conducted during hot weather; propellant sitting in the sunshine could have reached 60°C.)

Propellant temperatures were not originally measured because we knew of no instance where it was significant. Explosives such as Composition B will detonate at liquid nitrogen temperature, and nitromethane was checked over the temperature range in question with no affect on reaction time. However, in view of the nondetonability of NOS-365 at low temperatures, further tests were made wherein the propellant tank (on the impedance mirror) was warmed, using laboratory heating tape, to temperatures of 48-63°C. All such warmed charges showed evidence of detonation. Figure 6 is a typical streak photo. Note that the propellant charge is 305 mm long. There can be no doubt that detonation was occurring since the booster shock would be degraded and rounded after about 25 mm of travel through an inert liquid. In this case, the mirror was 38 mm thick, thereby allowing a longer observation time. The oscillating mirror elements show reaction for at least 9 microseconds.

Primary reflectivity loss has been an extremely annoying problem, but no solution for this has been found. It is not due to mirror degradation because covering the mirror with plastic sheeting has had no effect and this is known to prevent degradation at pressures to 30 GPa. Our best estimate is that the wave is so rough that mirror elements are turned to extinction. In this case, a much larger diameter

light bomb might have helped but such changes were outside the scope of our charter. If the mirror is indeed extremely rough, it provides further information on the detonation properties of the monopropellant.

The impedance mirror data showed a long reaction time in the propellant. Based on this, we have postulated that (1) NOS-365 is very insensitive, as we already knew, and (2) it has a critical run-up distance before reaching detonation assuming mild ignition. The impedance mirror test was originally designed to measure the induction and reaction time in explosives for the purpose of determining their intrinsic sensitivity. If we consider what is meant by the term sensitivity, it mainly reduces to the two elements of magnitude and time. Given two impulses of equal duration but different magnitude, a sensitive material will respond to the smaller magnitude impulse. Again, given two impulses of equal magnitude but different duration, a sensitive material will respond to the shorter duration impulse. The nature of an induction time is such that nothing can occur until this time is exceeded; the nature of a reaction time is such that an explosive reaction cannot build up to full power until it is exceeded. In this way, we can say that, if initiation pressures are comparable (as they were), NOS-365 with a 10+ microsecond reaction time is less sensitive than nitromethane with a 22 nanosecond reaction time. But, given a long reaction time, an unloading wave of any origin can easily quench the reaction, and detonation tests in thin walled tubes quickly produce unloading waves as the walls rupture. Additionally, unloading waves occur behind the booster shock due to the finite size of the booster. Reasoning of this sort led to the idea that massive confinement that would prevent all unloading waves might lead to detonation from a small stimulus. Confinement of this type is provided by a *gun chamber*. This, then, was the basis for conducting the tube tests.

TUBE TESTS

The tube test is done in a 0.9 meter long, upright mild steel tube having a 25 mm bore and 25 mm thick walls. The tube is plugged at the lower end by a 76 mm long tight-fitting steel plug welded in the tube. A detonator (RP-81 exploding bridgewire type) is lowered to the bottom of the tube and rests on the welded plug with lead wires running the length of the tube. The tube is filled to the top with propellant (remote operation). The detonator is confined by the steel tube and the weight of the propellant column. The tube is otherwise open at the top. Tested in this way, at an ambient temperature of 15-21°C, the steel tube is fragmented as shown in Figures 7a and 7b.

As mentioned previously, NWC had been conducting studies of bipropellants for use in the liquid propellant gun. Spark ignition tests of these bipropellants required that the material first be emulsified. A shaker arm having variable and controllable frequency was therefore fitted with a sample holder, electrodes and necessary wiring.

Although emulsification was not required for the NOS-365 monopropellant, this same shaker arm apparatus was later used for spark ignition tests of the NOS-365. Fastax camera coverage showed what appeared to be a reaction wave going through the propellant fluid which did not break the Lexan containers. Best estimates were that it was due to a reaction wave in the propellant's HAN component. It seemed possible that a 13-molar HAN solution would react in the tube test, and one such test was conducted. The explosion was accompanied by a brown cloud of NO_2 and yielded the fragments shown in Figure 8. Most of the broken edges were corroded, indicating that those breaks occurred during the explosion event when the HAN reaction products were vented across the fractures. Several breaks showed clean metal surfaces and we postulate that these breaks occurred during impact with the surroundings.

The fragments shown are primarily the brittle fracture type indicative of a low pressure. Additionally, these are large size fragments of the type one would expect from a ruptured pressure vessel. On the basis of this single test, we suspect that the HAN component of NOS-365 controls the sensitivity of the propellant although the full power output of the propellant depends on its further reaction with other components.

Ballistics Research Laboratory (BRL), Aberdeen, Maryland, personnel doubt that the tube test fragments show detonation at all since the shear zones are more shallow than they expected. We believe the depth of the shear zones indicates only that full power output is never achieved in a mild steel tube. This effect is, again, a function of the very long reaction time. We have performed a slight variation of the tube test which we believe shows this and, in addition, shows that NOS-365 does indeed have a critical run-up distance.

INDICATION OF CRITICAL RUN-UP

In two tube tests the confinement was increased by firing in 20 mm Mann barrels. The initiation was also strengthened by attaching a 2.5 gram tetryl pellet to the RP-81 detonator, which itself contains 850 mg tetryl. The length of the gun tube was 0.76 meter (30 inches) with a 25.4 mm long welded plug in the lower end (5 foot barrel cut in half). The bore was 20 mm. The dimension changes were considered to be minor. Real changes were the increased confinement of the 4340 steel and the more powerful initiation. The remains of such a test are shown in Figure 9. In this case, the shear zones extended nearly half-way through the 19 mm (3/4 inch) wall of the gun tube. This figure clearly shows that, despite the hard initiation, the detonation did not occur until the wave had propagated 0.3-0.38 meter (12-15 inches) from the booster. When venting occurs in any slow reaction, the pressure drops and the reaction is quenched. Despite the deep shear zones, it is questionable whether or not the propellant reached its full power output and one could certainly argue that it had not. The 20 mm gun tube tests clearly indicate that the mild steel test tubes fracture early in the reaction and vent the pressure before the reaction reaches completion. In the 20 mm gun tube, no fracture occurs at the booster end even though the inner walls are

blued from reaction heat. In the mild steel tube, the steel is indented at the end of the plug closure and clearly shows (Figure 7b) the result of having been in contact with the exploding propellant.

It is possible that an even heavier-walled gun tube would indicate the detonation run-up to be somewhat longer since it would hold together longer thus displacing the bulge further from the breech end. Whether or not the propellant is truly detonating, and whether or not the reaction has reached its full pressure potential, is somewhat academic. These questions could, of course, be answered with adequate instrumentation but we were only interested in a reasonable indication of whether or not the propellant could be controlled in a fail-safe manner in a real gun.

We believe the propellant can be controlled by making the gun chamber shorter than the critical detonation run-up distance. Although this might limit the gun caliber, it seems reasonable to expect that the caliber can be increased by changes in the propellant formulation. In view of the temperature effect shown in the impedance mirror tests, we expected some differences in the tube tests but this has not yet been documented. Several tube test variations have been proposed for future work. These include (1) tests on carefully debubbled liquid, (2) tests wherein the liquid contains deliberate bubbles of known size and placement, (3) critical run-up tests with a pseudo projectile in the bore, (4) ignition with a graded series of smaller detonators, approaching ignition with a spark, (5) tests with pyrotechnics ignition, (6) tests over a temperature range, and (7) tests wherein the reaction velocity and pressure are measured.

FRAGMENTATION FROM KNOWN DETONATIONS

Based on considerations of chemical composition alone, NOS-365 should be capable of developing a reasonably high detonation pressure. As has been mentioned, the long reaction time means that ultimate pressure is almost entirely dependent on confinement. As a comparison, two 20 mm Mann barrel sections were tested with known detonating materials both of which have nanosecond reaction times. These materials were Composition C-4 with a detonation pressure of about 24 GPa (3.5 million psi) and nitromethane with a detonation pressure of about 13 GPa (1.9 million psi). Fragmentation with these materials was considerably different than with NOS-365. Fragments from the high order detonations are shown in Figures 11 and 12.

In both known high order detonation cases, the rifling of the 20 mm barrel exerted an important degree of control in fragment formation. However, the depth of shear is not greatly different than that from NOS-365 (Figure 10). In both high order cases the barrel was shattered into smaller and more numerous fragments, but this is believed to be entirely due to reaction time differences. Pressure buildup is 1-2 orders of magnitude slower in NOS-365 than in Composition C-4.

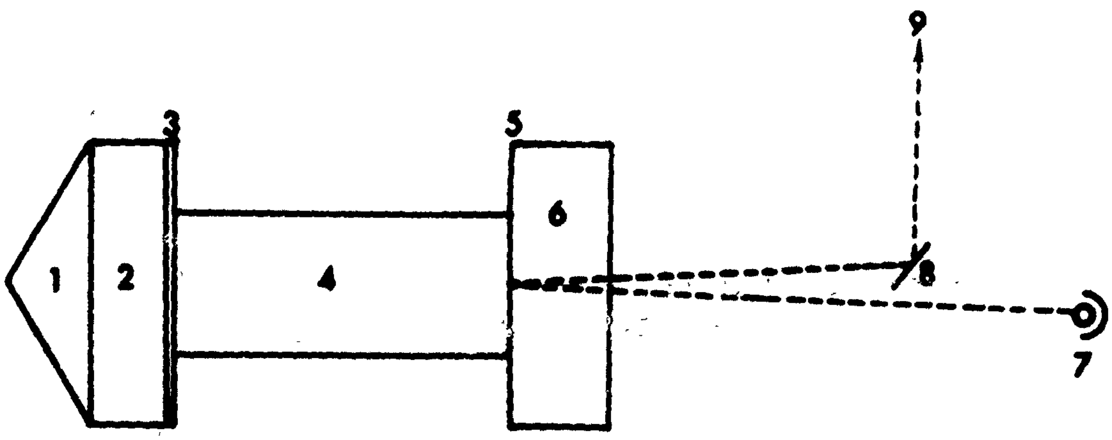
All the above described tests were tests of function. Tests were also performed to learn how NOS-365 can be safely handled.

SPILL TESTS

BRL, Aberdeen, tests showed that transition metal oxides catalyze the decomposition of NOS-365. The desert soil of the southwestern U.S. is rich in a magnetic iron oxide and, in addition, soil temperature under the summer sun is known to range as high as 77°C. Soil condition in other parts of the world where military action might occur could be at least as unfavorable. For this reason, under controlled conditions, a series of two types of spill tests were performed: (1) spills onto soil containing the naturally occurring magnetic iron oxide, and (2) spills onto rusty iron. For spills onto soil, the critical temperature above which reaction occurs rapidly is 60°C. For spills onto rusty iron, the critical temperature appears to be lower, about 50°C, although the fume-off reaction on one occasion occurred at 45°C after a 30 minute delay.

A test assembly with fume-off on soil is shown in Figure 13. In this case, local soil containing magnetic iron oxide was contained in an aluminum pan heated from the bottom by laboratory heating tape. The NOS-365, in a beaker above the soil pan, was also heated and the poured onto the soil (remote procedure). The pan and beaker heaters were turned off before the test to allow the temperature of both test components to equilibrate. The reaction shown in Figure 13 occurred after about 5 seconds of contact.

The above mentioned spill test on rusty iron at 45°C was performed under springtime conditions (air temperature 24.5°C and in full sunshine). The rusty iron under these conditions was at 45°C and might have gone higher with longer exposure to the sun.



Legend:

1. Plane wave lens
2. Composition B disc
3. Aluminum separator plate
4. Explosive tank
5. Saran covered reflective layer
6. Plexiglas mirror substrate
7. Argon-explosive flash
8. Turning mirror
9. To camera

FIGURE 1. Impedance Mirror Setup Schematic.

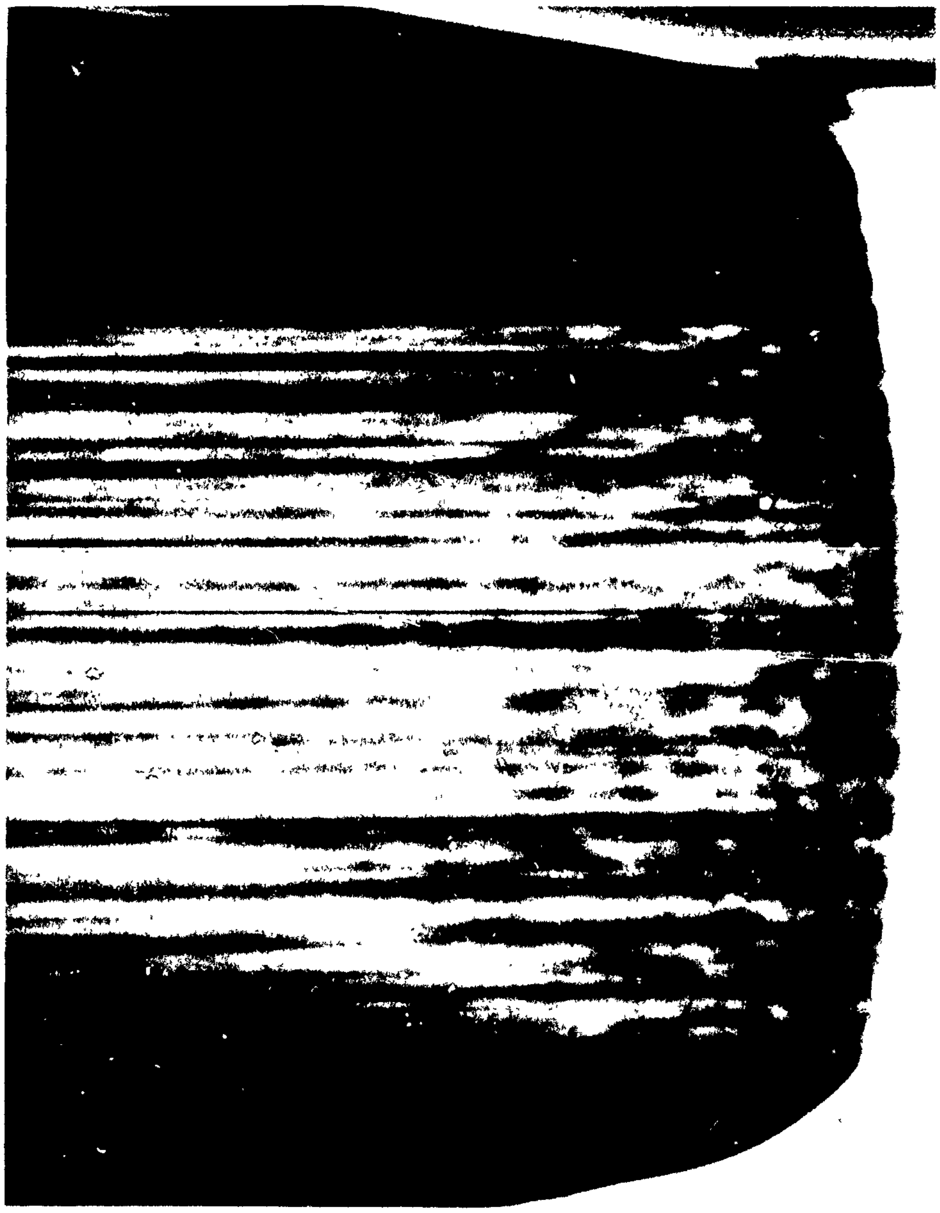


FIGURE 2a. Overdriven Wave in Diluted Nitromethane 60/NM, 40/Acetone.

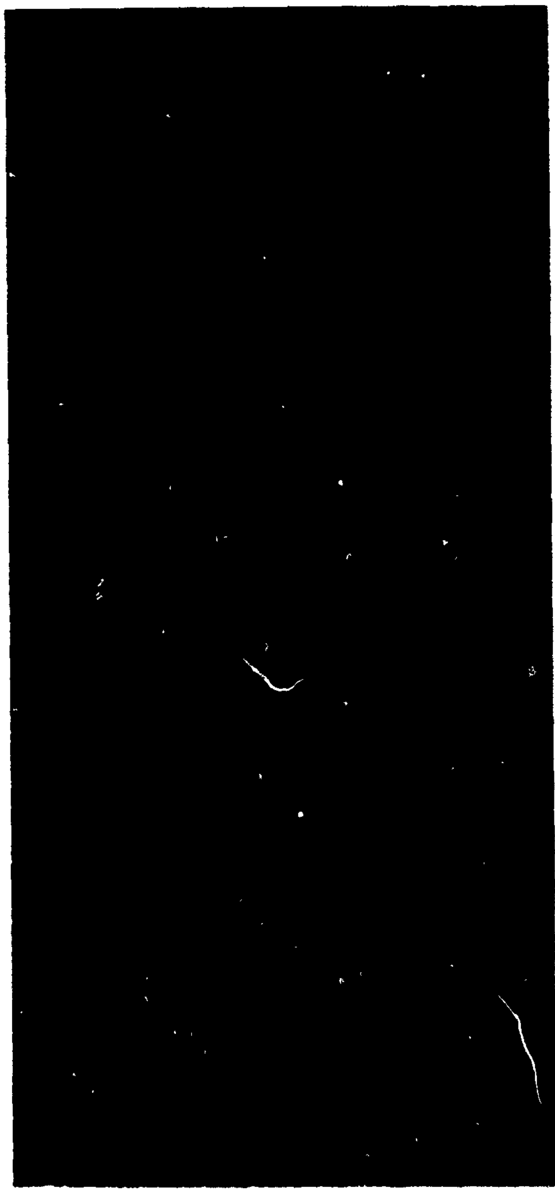


FIGURE 2b. Steady-State Wave in Diluted Nitromethane 90/NM, 10/Acetone Showing a Short Reaction Zone.

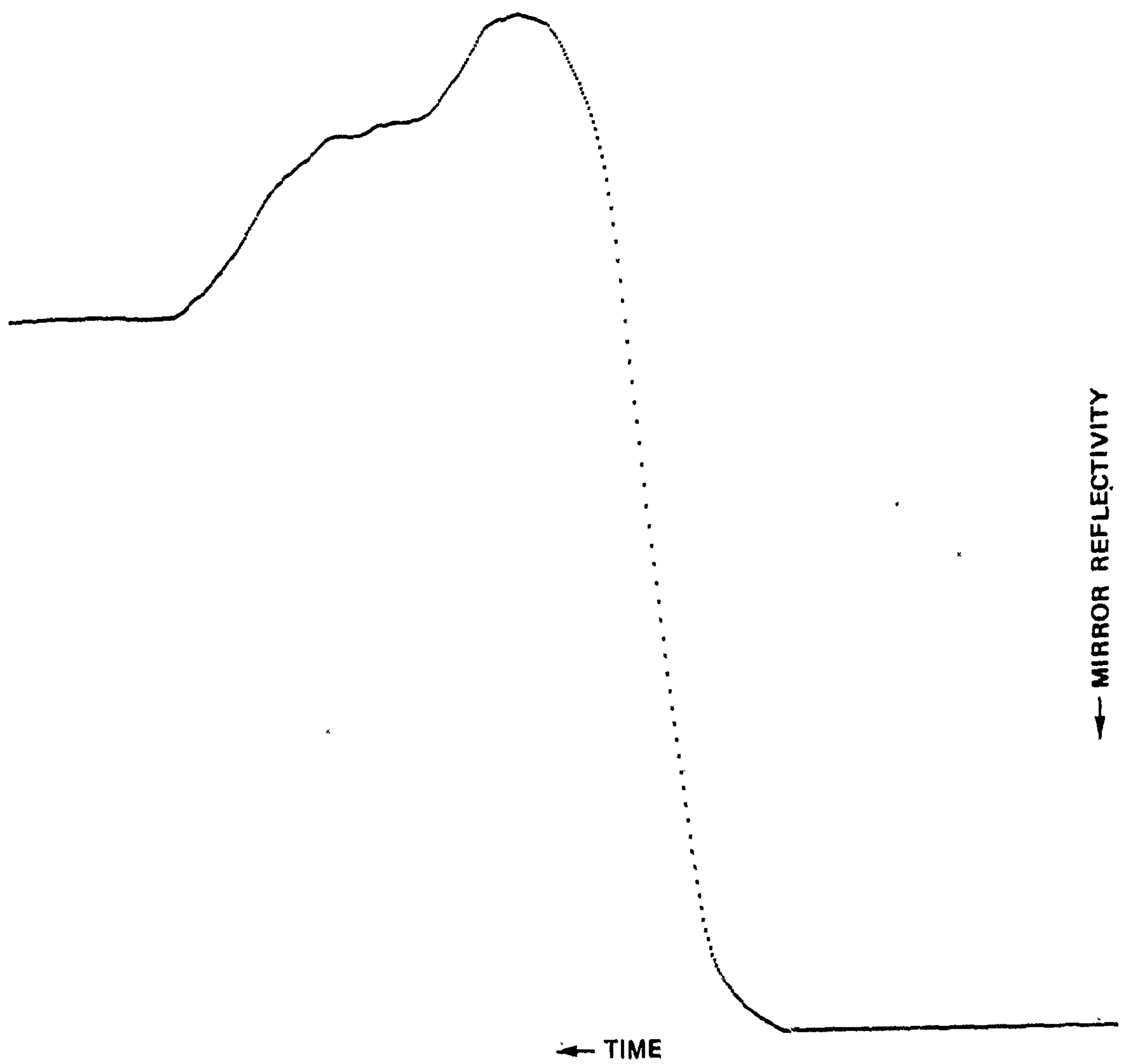


FIGURE 3. Photomultiplier Output Plotted Versus Time for a Record Such as Shown in Figure 2a.

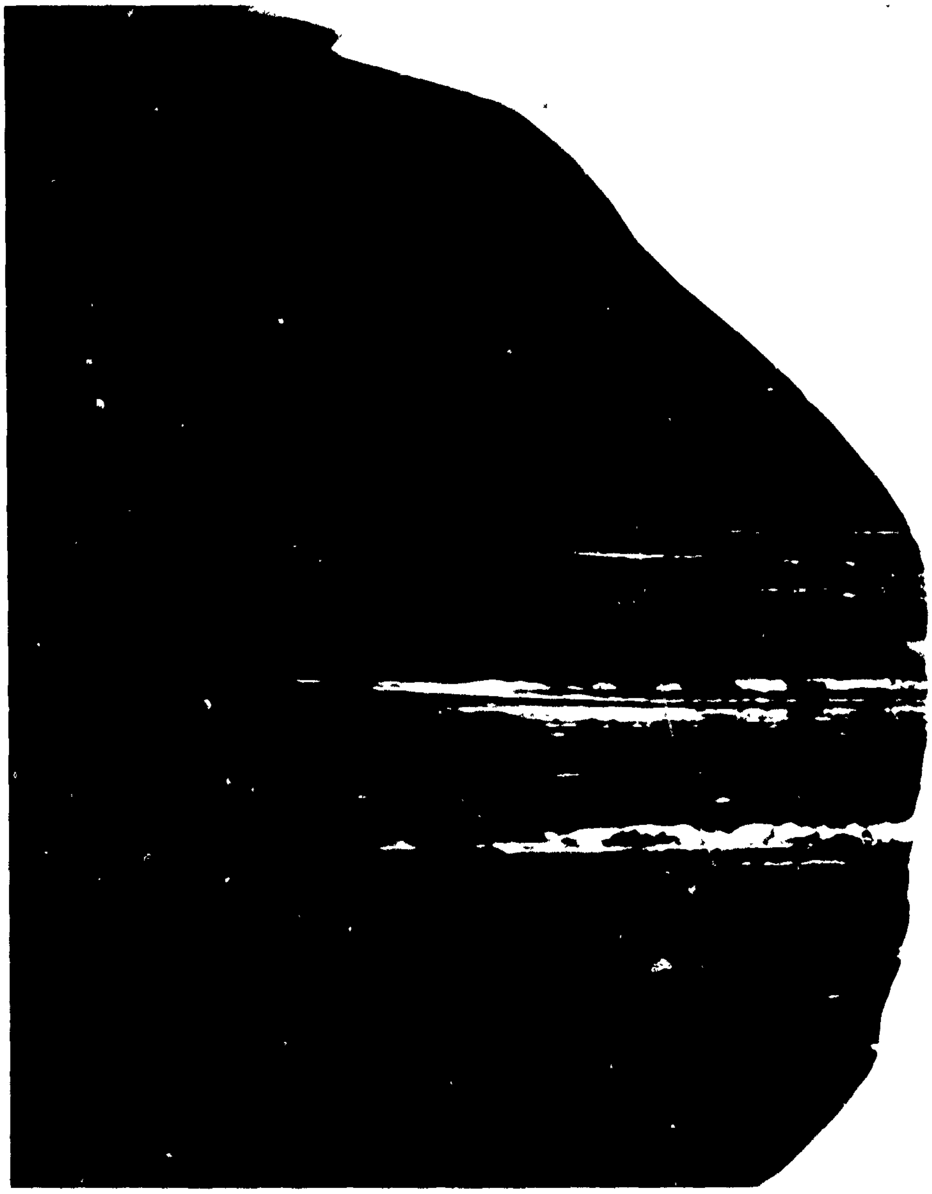


Figure 4a. Impedance Mirror Test ACS-222 (15 August 1976).

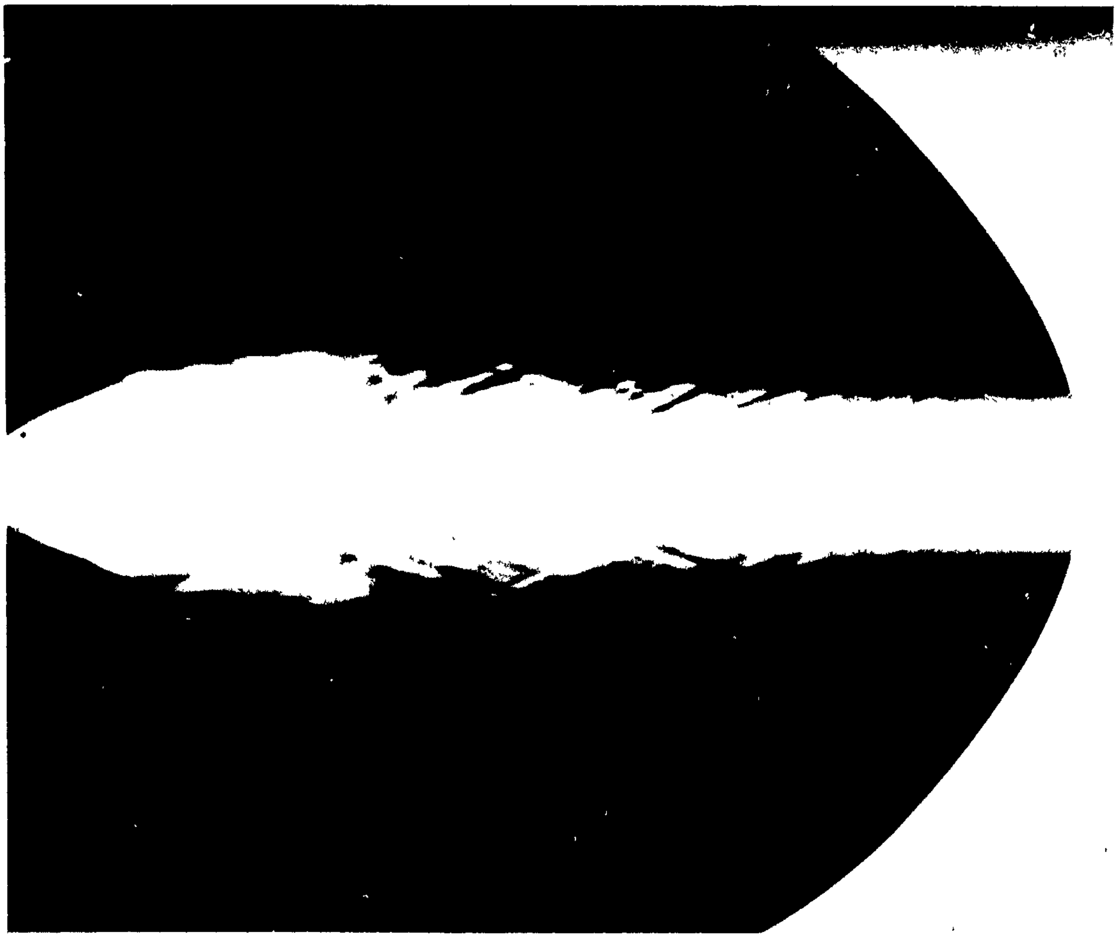


FIGURE 4b. Impedance Mirror Test ACS-218 (15 August 1976).

FIGURE 5. Impedance Mirror Streak Record NOS-365 at About 0°C.

FIGURE 6. Impedance Mirror Streak Photo of Detonation in NOS-365 at 54°C. Charge 305 mm long 95 mm ID. Standard P-40, Composition B booster.

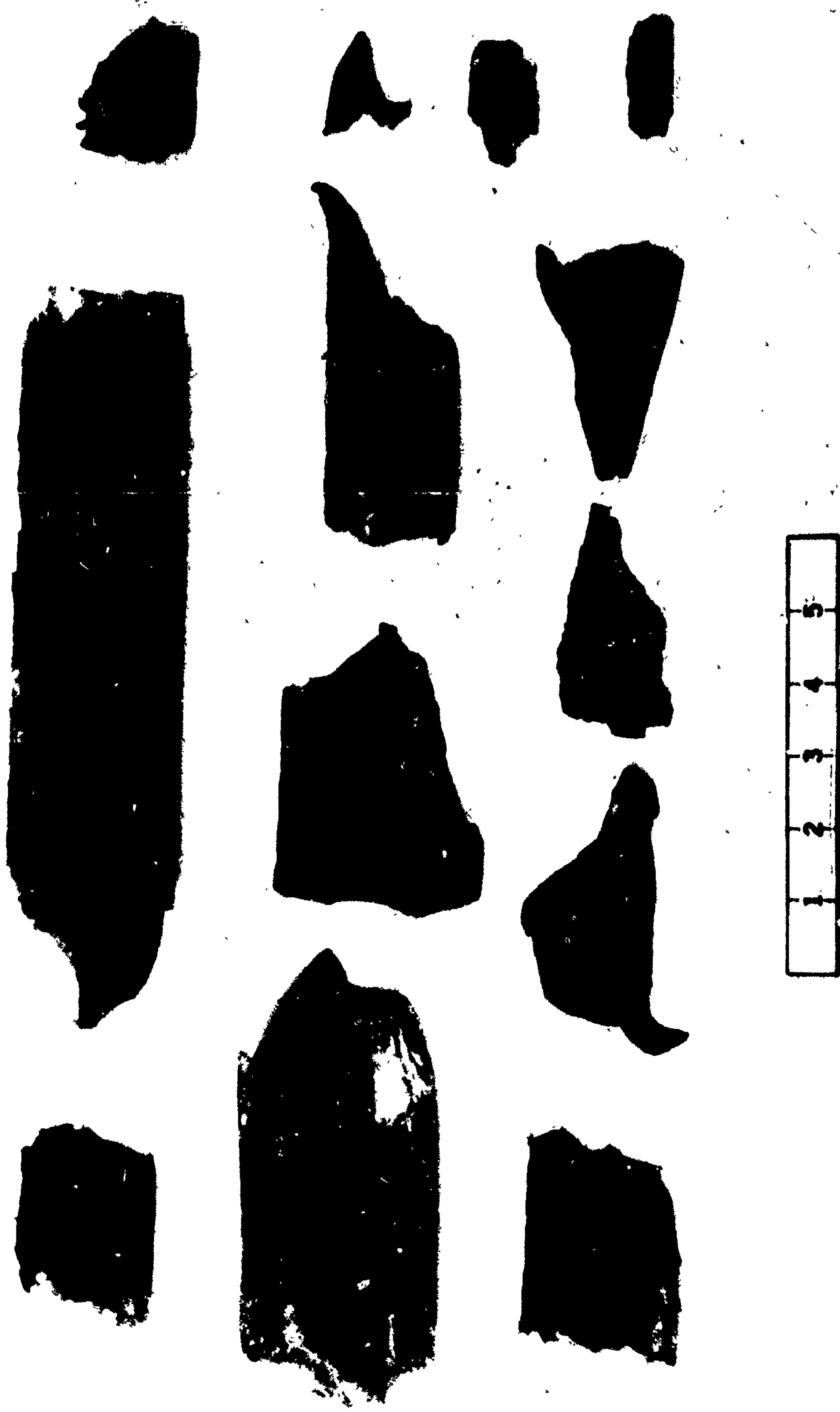


FIGURE 7a. Tube Test Fragments of Mild Steel from Detonation of NOS-365 (RP-81). (Scale in inches).

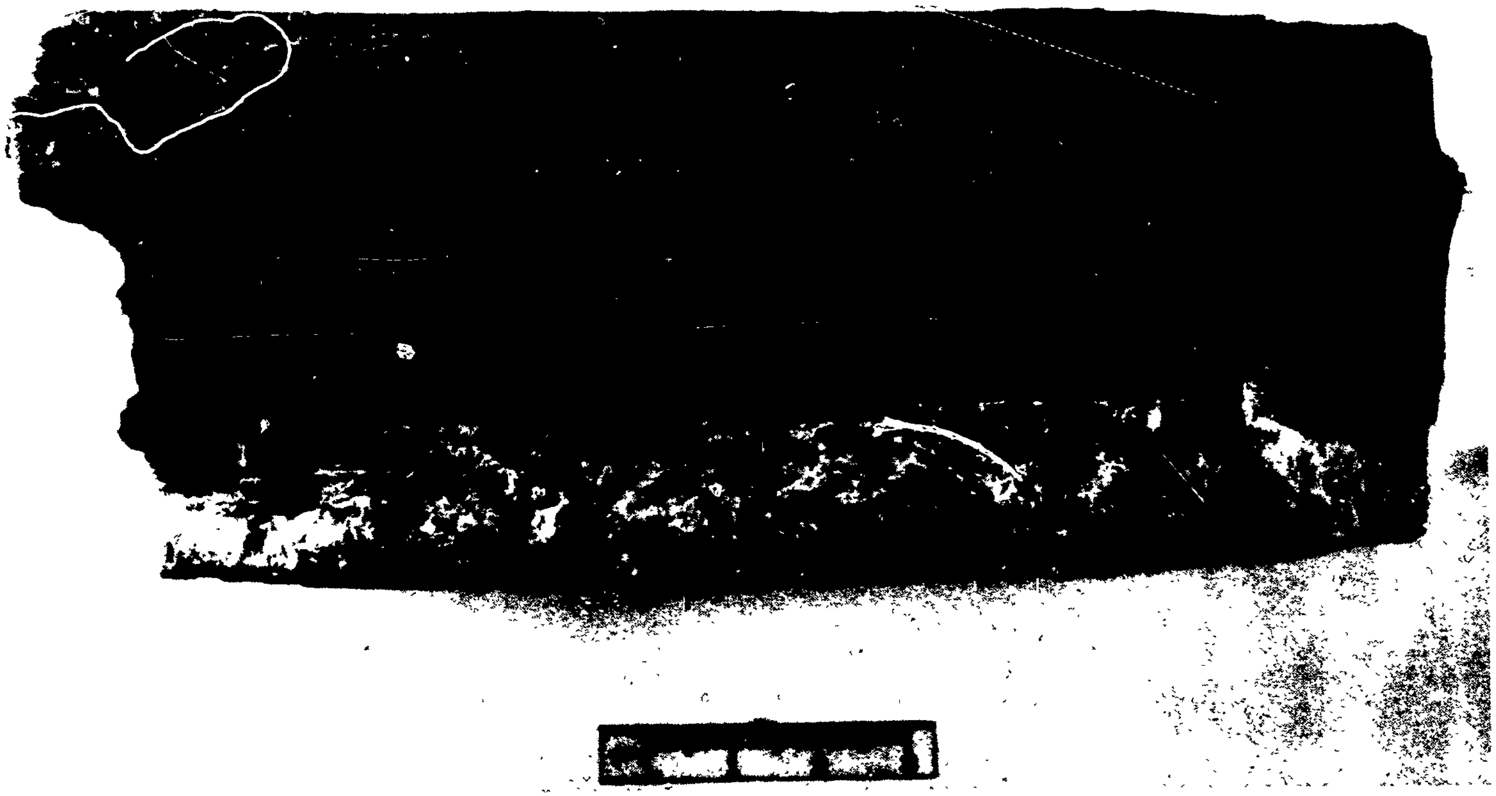


FIGURE 7b. Single Tube Test Fragment from Detonation of NOS-365 Showing Shear Fracture Beginning at Top of Steel End Plug. (Fracture in region filled by steel plug is brittle. Shear-checking in the bore is apparent. RP-81 initiation.)

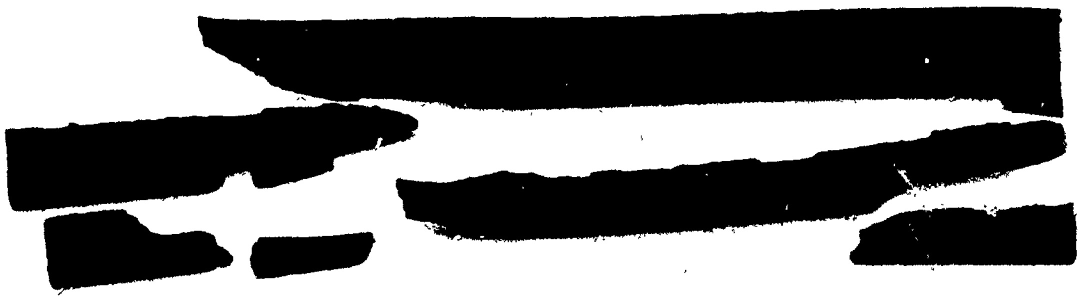


FIGURE 8. Tube Test Fragments from Detonating 13 Molar HAN Solution (RP-81 Initiation Test and Temperature About 15°C).

FIGURE 9. Section of 20 mm Mann Barrel Used in NOS-365 Tube Test. (Initiation by RP-81 plus 2.5-gram tetryl pellet. Detonation occurred 12-15 inches above point of initiation. Temperature about 21°C. Scale in cm.)

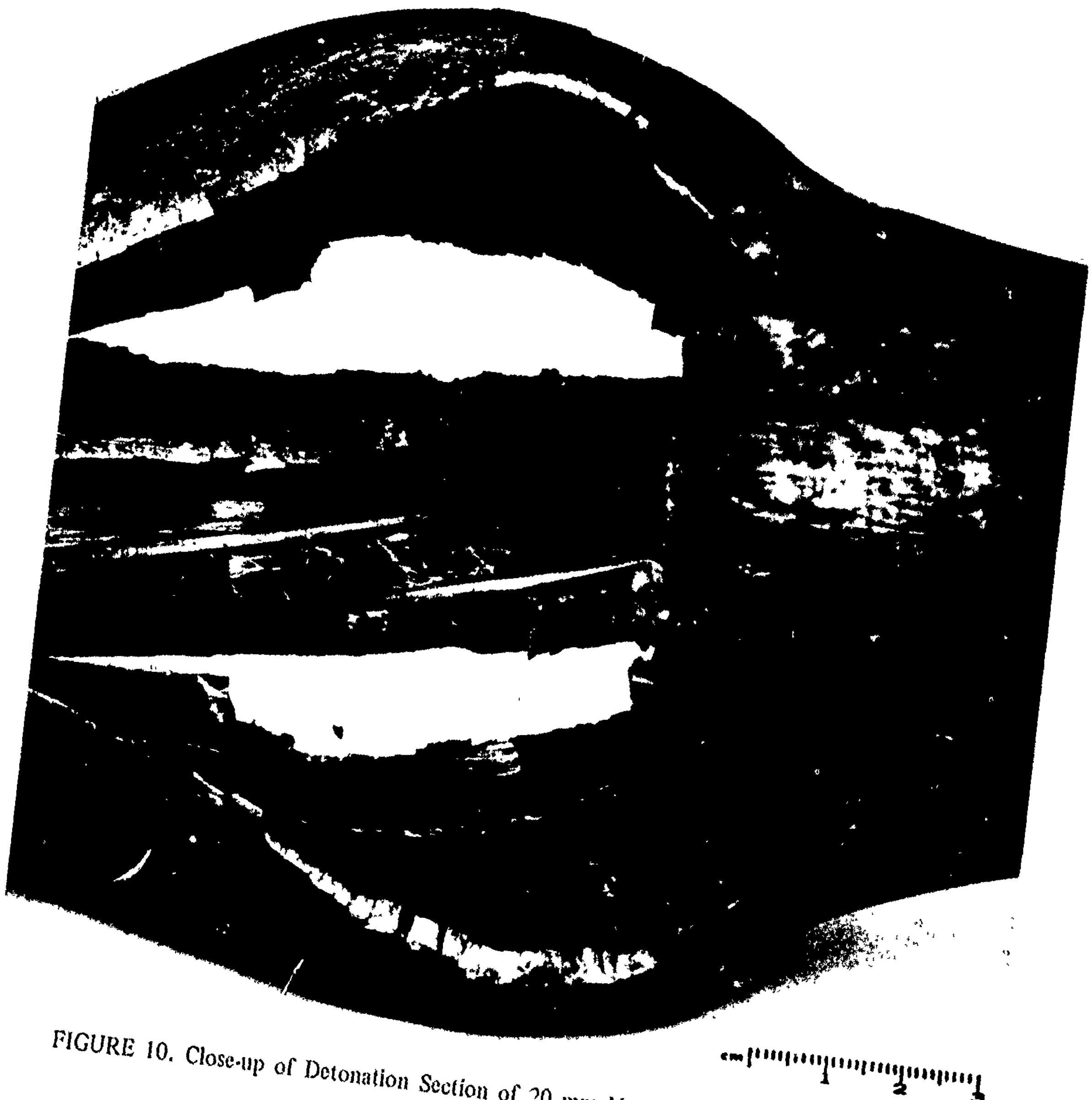


FIGURE 10. Close-up of Detonation Section of 20 mm Mann Barrel Shown in Figure 9.

FIGURE 11. 20 mm Mann Barrel Tube Test Fragments from Nitromethane (Scale in cm).

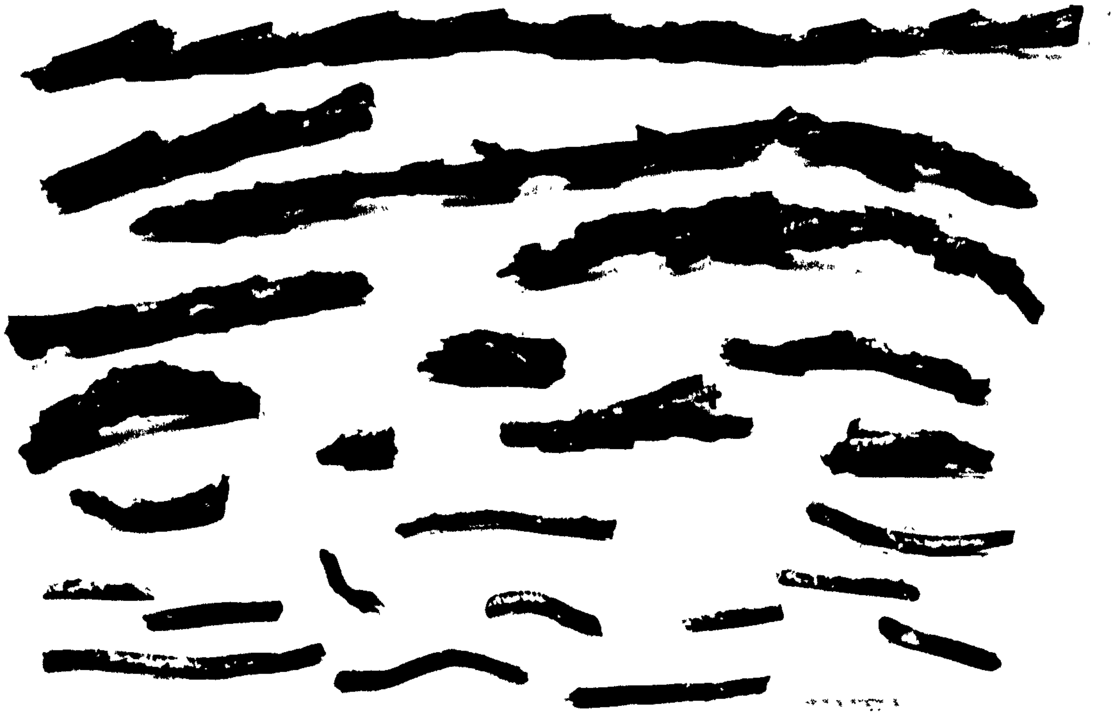


FIGURE 12. 20 mm Mann Barrel Fragments from Composition C-4 (Scale in cm).

FIGURE 13. Fume-off Test for NOS-365 at 66°C Spilled onto China Lake Soil at 66°C.

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1 Army Research Office, Durham (RDRD-ILP-CH, Info Proc. Office, Richard O. Ulsh)
1 Frankford Arsenal (J8100, Propellant Division)
1 Rock Island Arsenal (SARRI-LR, K. Whitwer)
3 Headquarters, U. S. Air Force
  AFRDPS, T. R. Ferguson, Jr. (1)
  DLFP, MAJ Baca (1)
  RDQRM, MAJ L. T. G. Lancaster (1)

1 Air Force Armament Laboratory, Eglin Air Force Base (DLOS-STINFO, N. H. Cox)
1 Air Force Rocket Propulsion Laboratory, Edwards Air Force Base (Technical Library)
1 Air University Library, Maxwell Air Force Base (AUL-SE)
1 Armament Development & Test Center, Eglin Air Force Base (XRC, T. O'Grady)
1 Deputy Director of Defense Research & Engineering (G. R. Makepeace)
1 Defense Advanced Research Project Agency, Arlington (TTO, Charles Lehner)
2 Defense Documentation Center
2 National Aeronautics & Space Administration
    Code RP (1)
    Technical Library (1)
1 Lewis Research Center (Librarian)
1 Lyndon B. Johnson Space Center (Code JM6, Technical Library)
1 Aeronutronic Ford Corporation, Newport Beach, CA (Technical Information Services Acquisitions)
1 Aerospace Corporation, Los Angeles, CA (library Acquisitions Group)
1 Allegany Ballistics Laboratory, Cumberland, MD
1 Battelle Columbus Laboratories, Columbus, OH (Stan Goddard)
1 Calspan Corporation, Buffalo, NY (Ed Fisher)
3 Chemical Propulsion Information Agency, Applied Phycics Laboratory, Laurel, MD
    Code ML, P. L. Nichols (2)
    Thomas W. Christian (1)
1 Flow Research, Inc., Princeton Combustion Laboratory Division, Princeton, NJ (Dr. Martin Summerfield)
1 Foster-Miller Associates, Inc., Waltham, MA (Engineers, Alve Erickson)
2 General Electric Company, Burlington, MA (Armament Systems Department)
    E. Ashley (1)
    N. Garland (1)
1 Hercules, Incorporated, Wilmington, DE (Technical Information Center)
1 Jet Propulsion Laboratory, CIT, Pasadena, CA (Library OP GR-Acq.)
1 Lockheed Missiles & Space Company, Sunnyvale, CA (Technical Information Center)
1 Los Alamos Scientific Laboratory, Los Alamos, NM (Code T-3, Dr. Don Burler)
1 McDonnell Douglas Astronautics, Huntington Beach, CA (Technical Library Service)
1 McDonnell Douglas Corporation, St. Louis, MO (Technical Library)
1 Northern Ordnance Division, FMC Corp., Minneapolis, MN (John Oberg)
1 Princeton University, Forrestal Campus Library, Princeton, NJ (Librarian)
1 Pulsepower Systems Incorporated, San Carlos, CA (L. Elmore)
1 R&D Associates, Marina del Rey, CA (Dr. Ray Edelman)
1 Rockwell International Corporation, Canoga Park, CA (Dept 086-306, Library)
1 Stanford Research Institute, Menlo Park, CA (H. B. Wilder, Jr.)
2 TRW Systems, Incorporated, Redondo Beach, CA
    Dr. E. Fishman (1)
    Technical Information Center (1)
1 Textron, Incorporated, Bell Aerosystems Co., Div., Buffalo, NY (Technical Library)
1 The Boeing Company, Seattle, WA (Kent Library, E. C. Vogt)
1 The Martin-Marietta Corporation, Orlando, FL (MP-30, Engineering Library)
1 Thiokol Corporation, Wasatch Division, Brigham City, UT (Technical Library)
1 United Technologies Corporation, East Hartford, CT (Acquisitions Librarian)
1 United Technologies Corporation, Sunnyvale, CA (Chemical Systems Division, Technical Library)
1 University of Utah, Salt Lake City, UT (Department of Chemistry, Dr. W. A. Guillory)
1 Vought, Incorporated, Systems Division, Dallas, TX (CH. Lib)